

# **YTTRIUM OXIDE BASED SURFACE COATING FOR SEMICONDUCTOR IC PROCESSING VACUUM CHAMBERS**

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## BACKGROUND OF THE INVENTION

## 1. Field of the Invention

## The present invention

The present invention relates to equipment utilized in the manufacture of semiconductor integrated circuits (IC) and, in particular, to utilization of a yttrium oxide ( $Y_2O_3$ ) coating on the anodized aluminum alloy components utilized in a semiconductor integrated circuit vacuum process chamber to improve corrosion resistance and erosion.

## 2. Discussion of the Related Art

Corrosion/erosion resistance is a critical property for parts used in IC fabrication vacuum chambers, where both corrosive chemistries and high-energy plasma bombardment reduce component lifetime and create contamination problems.

Anodized aluminum alloy is a primary material used in making components utilized in IC processing chambers. However, the high concentration of impurities contained in

conventional aluminum alloy causes formation of precipitates in the alloy which, in turn, cause internal cracks in the anodization layer. The integrity of the anodization layer is, thus, deteriorated and its corrosion 5 resistance is severely damaged. The impurity concentration (wt.%) of "conventional" aluminum alloy, such as 6061, is as follows: Mg=0.8-1.20; Cu=0.15-0.40; Zn=max. 0.25; Mn= max. 0.15; Fe=max. 0.70; Si=0.40-0.80; Others=max. 0.15

10 More recently, high purity aluminum alloy material has been developed, resulting in minimal internal cracking in the anodization layer. Current data shows that chamber parts made from high purity aluminum alloy materials perform much better than those made from conventional 15 alloy. "High purity" aluminum alloy means aluminum alloy with all impurities other than Mg being less than about 0.1 wt.% each, particularly Si, Fe and Cu.

20 Although high purity anodized aluminum alloy has a much better corrosion/erosion rate than the traditional anodized aluminum alloy, the anodization layer will still be attacked by the aggressive chamber environment after prolonged usage. The resulting need to replace parts reduces tool up-time and increases the cost of ownership.

25 Therefore there is always a need to continuously improve the lifetime of the aluminum alloy chamber components.

30 We are aware that yttrium oxide coatings have been used on anodized aluminum in the automobile and aerospace industries.

## SUMMARY OF THE INVENTION

5 We have found that applying a ceramic-based surface protective layer, a yttrium oxide ( $Y_2O_3$ ) coating, on the anodized surface of aluminum alloy chamber components improves the resistance of the anodized surface to corrosion and erosion by a factor of 5X over the anodized surface alone, particularly in the fluorine/oxygen plasma environment typically used in fabricating ICs.

10 A better understanding of the features and advantages of the present invention will be obtained by reference to the following detailed description and accompanying drawings that set forth an embodiment in which the principles of the invention are utilized.

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## BRIEF DESCRIPTION OF THE DRAWINGS

20 Fig. 1A is a SEM photograph showing a cross-section of a yttrium oxide coating on anodized aluminum alloy.

Fig. 1B is a SEM photograph showing a cross-section of a yttrium oxide coating on anodized aluminum alloy at higher resolution than the Fig. 1A SEM photograph.

25 Figs. 2A and 2B are SEM photographs showing, respectively, as-coated and after surface finished anodized aluminum alloy coated with yttrium oxide in accordance with the present invention.

## DETAILED DESCRIPTION

Prototype vacuum chamber components made from high purity anodized aluminum alloy (e.g., chamber liner, cathode liner and door) were coated with various candidate materials for testing with respect to erosion rate. A final chamber test with coated parts was then conducted to verify the performance of the coating. The result of chamber tests showed that plasma spray coated  $Y_2O_3$  had the lowest etch rate among all tested materials.

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Anodization of the aluminum alloy can be in accordance with the disclosure of commonly-assigned and co-pending U.S. patent application Serial No. (not yet available), filed February 8, 2002, titled "Halogen-Resistant Anodized Aluminum for Use in Semiconductor Processing Apparatus" (Docket No. AM-6846) which application is hereby incorporated by reference in its entirety to provide additional background information regarding the present invention.

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More specifically, high purity 99.95% yttria was sprayed on coupons made from anodized high purity aluminum alloy material utilizing commercially available plasma spray coating techniques. Other techniques for applying the coating, e.g., chemical vapor deposition (CVD) and physical vapor deposition (PVD), are also appropriate.

Although the invention may be practiced utilizing conventional aluminum alloy, utilization of high purity aluminum alloy enables application of a barrier for the applied coating.

The typical coating thickness is 5-7 mils. Important physical properties of the  $\text{Y}_2\text{O}_3$  coating are listed as follows:

5	Composition	> 99.9% semiconductor Grade $\text{Y}_2\text{O}_3$
	Porosity	1 - 2% max.
	Density	5.0 g/cc
	Hardness	> 500 HV 0.3 (by cross-section)
10	Ra ( $\mu\text{in}$ )	120
	CTE	8 - 9 $\mu\text{m}/\text{m}/^\circ\text{C}$
	Thermal Conductivity	8 - 12 W/m. $^\circ\text{K}$
	Coating Adhesion	3700 psi (on bare Al substrate) 7100 psi (on anodized Al substrate)
15	Dielectric Constant	6-8 (ASTM D-159)

Fig. 1A shows the cross-section of an as-coated sample. Fig. 1B shows a similar SEM cross-section taken at higher resolution. The Fig. 1A/1B photographs clearly show that the coating layer was dense, with all pores being isolated from each other. The maximum porosity was determined to be less than 1-2% based on the apparent metallographic method. The  $\text{Y}_2\text{O}_3$  coating to high purity aluminum alloy substrate interface is intact. No delamination or other interfacial defects, such as voids and cracks, are observed.

It should be understood that up to about 10%  $\text{Al}_2\text{O}_3$  can be combined with the  $\text{Y}_2\text{O}_3$  to provide a  $\text{Y}_2\text{O}_3$ -based coating with improved hardness and breakdown voltage characteristics.

Electrical properties were tested per ASTM standards in order to satisfy minimally required electrical properties for plasma chamber components. The results are listed below:

Breakdown Voltage of $Y_2O_3$ Coating (V)				
	total voltage	voltage per mil		
5	anodization (1 mil)	1,533	1,533	
	$Y_2O_3$ on bare Al (6 mil)	5,232	872	
	$Y_2O_3$ on anodized Al	5,894	843	
Volume Resistivity (ohm-cm)				
	25°C	60°C	150°C	
10	anodization (1 mil)	7.4E15	4.1E15	4.9E15
	$Y_2O_3$ on bare Al (6 mil)	1.5 E15	6.0E14	7.8E13
	$Y_2O_3$ on anodized Al	9.8E14	4.1E14	3.4E13

The total breakdown voltage was found to be  $> 5$  kv for a 6 mil coating layer, which was well above the 1kv criteria for a conventional anodization layer. The volume resistivity was also high enough to meet typical process requirements.

One major concern for the spray coated part is surface loose powders which were applied to the substrate during the very last spraying cycle. If these particles are not completely removed by a final finishing/cleaning process, these powders will have a high chance of coming off from the part's surface due to dynamic chamber conditions (erosion, corrosion, and thermal cycle). Once released to the chamber interior, they will cause a severe particle contamination problem.

To assure that no such loose particles are left on the yttrium oxide coating surface, the following methods are used.

First, a light mechanical finish is performed as part of the coating processes by manually holding a grinding tool over the as-coated surface, using silicon carbide (SiC) as the grinding medium. Fig. 2A and 2B are scanning electron micrographs that show as-coated and after surface furnished surface, respectively, clearly demonstrating that, after coating, the surface is very rough, with many powders

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and nodules loosely attached to the surface. However, after finish, the coating surface is dense and free from loose particles.

After coating and surface finish, the coated parts are subjected to a standard cleaning procedure for chamber components that includes a CO<sub>2</sub> snow gun clean followed by a deionized (DI) water ultrasonic rinse performed at room temperature for about 15 minutes. All parts are then verified as particle-free by a Dryden QIII tool.

Cleanliness of the coating layer is another subject which requires close monitoring. Contamination can be introduced to the coating layer from various sources: raw powders, spray gun, process gases, and uncontrolled environment.

In the cleanliness study, the well-known Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) technique was used to determine the impurity levels in the coating layer. Table 1 below shows the impurity data of coating coupons. Two types of samples were used: after coating/finish from the coating vendor and after final clean from the cleaning vendor. Both samples were analyzed at surface and sub-surface for comparison. Each scan contained 60 pulses with 20  $\mu\text{m}$  dia. spot size each pulse. The penetration depth was 1-2  $\mu\text{m}$ . Sub-surface was achieved by pre-ablated top 1-2  $\mu\text{m}$  layer away before testing.

There was some impurity difference between surface and sub-surface; in general, the sub-surface was found to be cleaner than the surface. Final clean reduced the coating impurity levels, indicating that the final clean process was effective to remove contaminants from the surface.

	after final clean		after coating and polish	
	surface	sub-surface	surface	sub-surf
Li	< 0.1	< 0.1	< 0.1	< 0.1
Be	< 0.1	< 0.1	< 0.1	< 0.1
Na	6	< 1	< 1	< 1
Mg	7.9	10.0	6.7	3.2
Al	123	70	415	213
Ca	< 1	< 1	< 1	< 1
Ti	12.4	3.6	60.0	8.0
Cr	7	7	1	< 1
Mn	< 1	< 1	1	11
Fe	1	< 1	< 1	60
Ni	1.6	1.0	0.3	0.1
Zn	75.1	63.4	88.0	19.9

Table 1:  $\text{Y}_2\text{O}_3$  coating impurity analysis (ppm)

It should be understood that various alternatives to the embodiments of the invention described herein may be employed in practicing the invention. Further example, as discussed above, it should be understood that in addition to high purity 99.95% yttria, any yttria based coating will provide erosion/resistance enhancements over anodization alone. Thus, it is intended that the following claims define the scope of the invention and that methods and structures within the scope of these claims and their equivalents be covered thereby.

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